FLIGHT INSTRUMENT

The MEPHISTO apparatus is shown on the cover mounted on its bridge-like carrier in the space shuttle cargo bay and schematically in the logo. MEPHISTO will simultaneously process three stationary, rod-shaped samples (5.9 mm in diameter and almost 900 mm long). All three samples will receive identical heat treatments. The two furnaces are heated to a predetermined temperature. The maximum operating temperature is 900 °C. One furnace is mounted on a sliding mechanism that allows it to move back and forth. As the mobile furnace travels toward the fixed one, the sample solidifies. As it moves away from the fixed furnace, the sample melts. Several melting and solidification cycles will be performed under different conditions during the mission. The measurements made in space will be virtually free from the gravity-induced complications experienced on Earth. Researchers will compare data from space-based and ground-based experiments to better understand solidification and the effect of gravity on it.

facets exposed to the liquid during growth to be determined, but without solutal and/or thermal interference from other grains. Peltier demarcations in the processing zone outside the capillary portion of the sample will provide information on the shape of the solid/liquid interface in the presence of solutal/thermal interactions with adjacent orientations.

The third (quenching) sample (fig. 3(c)) will be used to track external furnace movement by measuring the resistance change across the sample. A 2-mm-inner-diameter capillary tube will be incorp-orated into the design to conform with the other two samples so that they will be directly comparable.

The Seebeck and Peltier samples will each contain two thermocouples spaced within the processing zone.

SPACE EXPERIMENTS

The flight experiment will be remotely controlled from the ground at the Payload Operations Control Center (POCC) at the NASA Marshall Space Flight Center near Huntsville, Alabama. Electromagnetic signals will be sent to the experiment directing its operation. Data from the experiment will be transmitted back to the POCC. Figure 4 schematically shows the proposed flight experiment plan. The flight plan consists of five steps: full melting, slow solidification, Seebeck measurements, Peltier pulsing, and quenching. All three flight samples will be melted and solidified simultaneously, thus providing identical thermal treatment. However, the Seebeck measurements, the Peltier pulses, and the quenching will each be made on separate samples. The processing zone of each sample consists of two portions: one with the inner capillary tube isolating a single grain and the other portion with its multiple grains. The steps in the flight experiment plan are described next.

Full Melting

The central 484-mm-long section of each sample is melted and the temperature is held constant.

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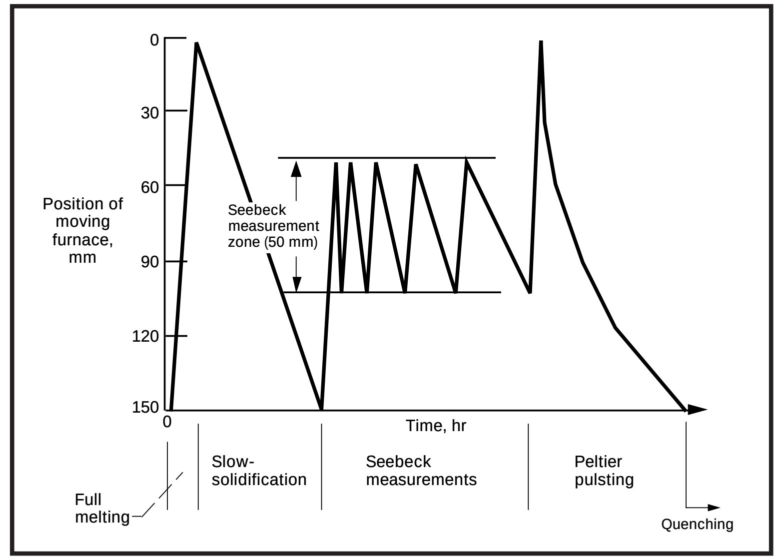


Figure 4.—Proposed MEPHISTO flight experiment plan.

Slow Solidification

A small portion (150 mm long) of each molten section is resolidified at a very slow rate (about 0.1 mm/min). This procedure establishes a uniform microstructure under microgravity conditions and provides an opportunity to conduct Seebeck measurements to be used as reference standards. Measurements obtained during subsequent tests at higher velocities will be compared with these standards.

Seebeck Measurement

A 50-mm length of the sample, within the 150-mm length used in the slow solidification step, is selected for the Seebeck measurements. The selection is based on that 50-mm length giving the "best" Seebeck signal (i.e., having the fewest imperfections and the most uniform composition). The measurements are made during solidification and remelting at five interface velocities, from 0.11 to 1.6 mm/min. The Seebeck measurements allow the detection, in real time, of changes in the interface microstructure.

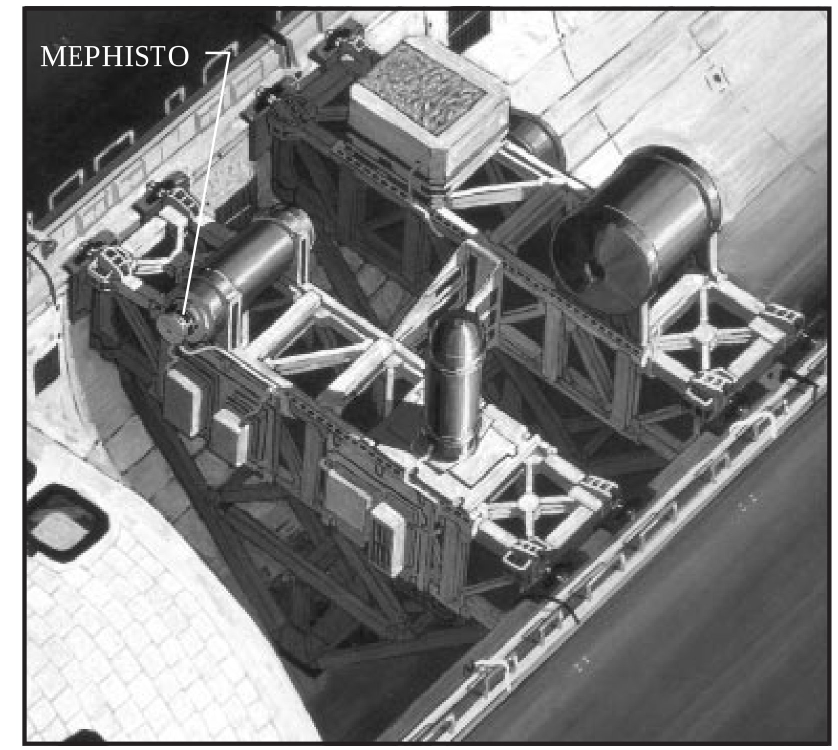
Peltier Pulsing

At the beginning of Peltier pulsing the samples are first remelted to the full extent of furnace travel (150 mm). They are then sequentially solidified at the six interface velocities, from 0.11 to 2.4 mm/min. At each velocity 10 Peltier current pulses, each with an amplitude of 50 A/cm^2, are made on one of the samples. This procedure will show the relationship between interface shape and interface velocity.

Quenching

The Seebeck and Peltier steps can be repeated. At the end of the experiment the final 2 cm of the third sample is cooled very quickly and then analyzed to determine the concentration of tin in the liquid prior to quenching.

In-Situ Monitoring of Crystal Growth Using MEPHISTO



United States Microgravity Payload

National Aeronautics and Space Administration

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NASA Headquarters
Office of Life and Microgravity Sciences and Applications
Microgravity Science and Applications Division

NASA Lewis Research Center
Space Experiments Division
Materials Division

INTRODUCTION

The In-situ Monitoring of Crystal Growth Using MEPHISTO Experiment is a cooperative American and French investigation of the fundamentals of crystal growth. MEPHISTO is a French-designed and -built materials processing furnace. Its name is the French acronym for Materiel pour L'Etude des Phenomenes Interessant la Solidification sur Terre et en Orbite (Materials for the Study of Interesting Phenomena of Solidification on Earth and in Orbit). The experiment will fly as part of the fourth United States Microgravity Payload (USMP–4) aboard the space shuttle *Columbia.*

Bismuth with small additions of tin will be studied in this experiment. These materials have a strong tendency to form faceted (flat faced) crystals when they solidify. Solidification (also called freezing) is the process where materials change from liquid (melt) to solid. An example of solidification is water changing into ice. These studies will extend the experiments conducted on USMP–2, mission STS–62 and also aboard *Columbia*, which flew March 4 to 18, 1994. The results from these two sets of experiments will be of fundamental importance to the manufacturing of metal alloys and materials for solid-state components, such as integrated circuits and electronic equipment. Examples include advanced medical equipment, computers, and home electronics.

SCIENCE

Chemical nonuniformities and physical imperfections significantly lower the quality of semiconductor crystals grown on Earth and thus degrade the performance of electronic devices used in industrial, military, and home electronic equipment. The crystal growth process is complicated by gravity. These complications are significantly reduced in low gravity, making it easier to understand the growth process. Improved understanding will influence the development of techniques for growing higher quality crystals on Earth.

A number of techniques can be used for growing crystals. The method used in this experiment is known as directional solidification. In this process the molten sample in the furnace is cooled at one end; it begins to solidify as the temperature of the liquid falls below the melting point. As cooling continues, the boundary between the solid and liquid material (the solid/liquid interface) moves from one end of the sample toward the other–hence the term "directional solidification." It is important to know the position of the solid/liquid interface and how fast it moves during crystal growth. Local conditions at the interface largely determine the rate at which the crystal grows, the mechanism by which it grows, and its structure and chemical composition. These factors strongly influence the quality of the crystal and its usefulness in electronic devices. In particular, supercooling at the solid/liquid interface is a key variable during growth.

> "Supercooling" describes the condition in which a liquid cools to below its freezing point before solidifying. The level of supercooling refers to the difference between the temperature of the solid/liquid interface and its normal freezing temperature.

These experiments use a novel technique to measure interface supercooling directly, in real time during growth, and without interfering with the growth process. The basis of this technique is the Seebeck thermoelectric effect. Figure 1 schematically shows this measurement principle. A voltage across the sample results from the difference in thermoelectric conditions exhibited by the solid and the liquid at their interfaces. One interface is moving and cooler; the other is at the equilibrium melting point and is stationary. The supercooling at the moving interface in the mobile furnace (see Flight Instrument section on flap) can be calculated from this voltage. Bismuth and tin have relatively large Seebeck signals, making them good candidates for use in MEPHISTO.

A key aspect of the crystal growth process is defining the growth conditions leading to stable microstructures (the structure of the crystal when viewed under a microscope). The microstructure of a crystal depends, in part, on the shape of the solid/liquid interface when the crystal was produced. When the interface is planar, a more uniform and featureless structure is produced. When it is cellular, a beehive-like structure (but less regular) is produced. When it is dendritic, a complicated structure is produced. The planar structure has the fewest defects (imperfections) and the most uniform composition. The rate at which the crystal grows and the temperature distribution in the liquid near the interface affect the interface shape and thus the microstructure. Measurements of these conditions are an important part of the experiment. It is not possible to view the microstructure at the interface directly in real time, but it is possible to determine the interface shape as it was at any time during

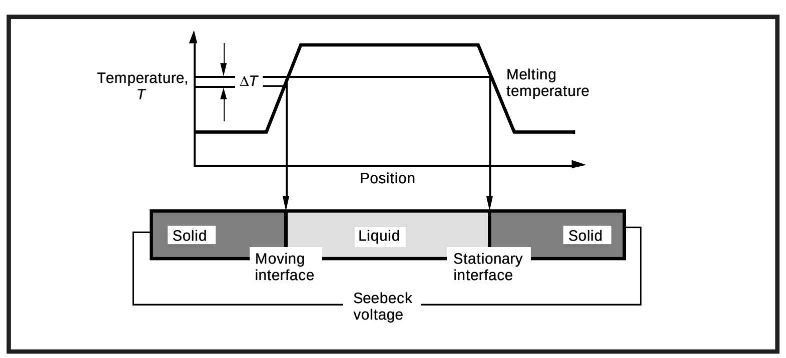

Figure 1.–Equivalent Seebeck circuit for measuring temperature of solid/liquid interface.

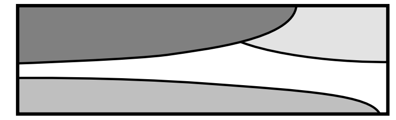

Figure 2.–Schematic showing three grains (single crystals with different orientations) typically contained in 6-mm-diameter longitudinal cross section of bismuth-tin directionally solidified samples from flight experiment aboard USMP–2.

growth. The technique for doing this is called Peltier marking. An electric current is pulsed through the sample. The current pulse causes a momentary change in the local chemical composition, which "outlines" the interface shape at that moment. That outline remains within the sample, unless it is remelted, and can be revealed later when the sample is cut, polished, etched, and examined under a microscope.

The experiments conducted on USMP–2 yielded a large amount of information concerning growth rate, interface shape, and interface supercooling. The bismuth-tin crystals used in these experiments were polycrystalline (i.e., they contained multiple grains). The grain boundaries contribute to the Seebeck voltage. This contribution must be accounted for in interpreting the Seebeck data. Further, bismuth is an anisotropic crystal, meaning that the properties of the crystal depend on the direction in which they are measured. The thermophysical properties of the crystal, including the Seebeck coefficient, are also orientation dependent. The experiment would have been simplified if single-crystal samples of bismuth-tin had been used. But it is nearly impossible to grow single crystals (no grain boundaries) of bismuth-tin alloys to the dimensions required for the MEPHISTO apparatus. For the experiments on USMP–4 the problem of single-crystal versus polycrystalline samples has been resolved by a clever experimental design conceived after microstructural analysis of the crystals grown during USMP–2. The researchers observed that the 6-mm-diameter cross section of the sample typically contained three or fewer grains (see fig. 2). The concept is to isolate one orientation (i.e., one grain) by incorporating a quartz capillary tube

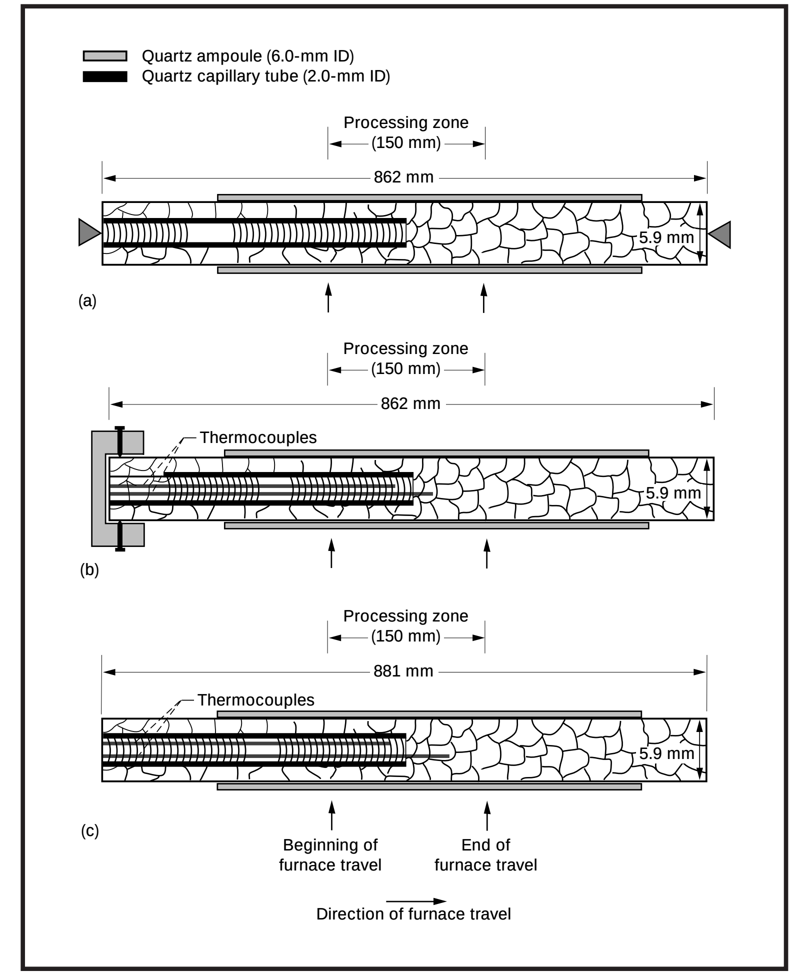

Figure 3.–Schematics of samples. (a) Seebeck; (b) Peltier; (c) quenching.

extending halfway through the 150-mm-long processing zone of the larger diameter sample. The initial series of microgravity measurements will be made on the sample inside the capillary tube; subsequent measurements will be made on a polycrystalline microstructure inside the main quartz ampoule (see fig. 3). Figure 3(a) shows the concept drawing for the Seebeck sample. The outer quartz ampoule is 6 mm in inner diameter by 10 mm in outer diameter, and the inner quartz capillary tube has a 2-mm inner diameter. That portion of the sample inside the capillary tube is a single crystal electrically isolated from the surrounding solid. By using this concept the researchers will be able to obtain Seebeck signals from the single-crystal interface exposed to the liquid column.

Similar to the Seebeck sample the design of the Peltier sample (fig. 3(b)) is again based on the solidification of an isolated orientation during the first half of microgravity processing. A 2-mm-inner-diameter quartz capillary tube will extend halfway through the processing zone of the larger diameter sample. Unlike the Seebeck case the sample in the Peltier capillary tube will be electrically connected to the surrounding sample at its end. The Peltier demarcations will allow the spatial orientation of the

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